Dynamic restoration of the ferrite and austenite phases during hot compressive deformation of a lean duplex stainless steel

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ABSTRACT

The active restoration processes within the ferrite and austenite phases were investigated in a Ni-free duplex stainless steel with the 75/25 austenite/ferrite ratio performing compression tests over the wide range of temperature and strain rate (700–1000 °C and 0.001–0.1 s⁻¹, respectively). The substructural features under different Zener-Hollomon parameters (Z) were examined using the electron backscatter diffraction analysis. A significant portion of flow softening was attributed to the active softening mechanisms in the continuous network of ferrite phase. The ferrite was softened through continuous dynamic recrystallization (CDRX) at high Z values, but the dynamic recovery (DRV) surrounded CDRX at low Z parameters. Interestingly, the discontinuous dynamic recrystallization (DDRX) mechanism was testified by increasing temperature and strain rate, i.e. at medium Z values. DDRX occurred by the growth of high-angle subgrains near the austenite/ferrite interfaces. With decreasing Z values, an increase in subgrain and grain sizes was observed. Contrarily to ferrite, the austenite phase was softened mostly through the dynamic recovery mechanism. At higher Z values microband arrays were formed in austenite holding specific character resembling to band-like misorientation gradients. However, under the lower Z values, a small fraction of recrystallized grains was formed through the CDRX/DDRX mechanisms. This was justified considering the lower strain accommodation in the austenite phase during deformation of duplex stainless steel.

1. Introduction

Duplex stainless steels (DSSs) involving austenite and ferrite phases demonstrate good combinations of strength, ductility and corrosion resistance since they benefit from the single phase counterparts, i.e. corrosion resistance of ferritic SS and mechanical properties of austenitic SS. The conventional DSS typically contains 18–26 Cr, 3–8 Ni, 2–5 Mo and 1–2 Mn (in wt.%) [1]. Not only does Mn addition enhance essential wear resistance but it increase the nitrogen (N) solubility as well. In addition to the conventional DSS, by the advent of developing the cost-effective lean alloy systems, the new generation of DSSs harnessing N have been presented. They are produced by substituting the costly alloying elements such as Ni and Mo with N and Mn [2–4]. Accordingly, this can be virtually related to a multiple effect of N, austenite stabilizer, on enhancing the pitting and/or crevice corrosion resistance, and strength. Hence, the Mn-N-bearing DSS without Ni content has received attention as a lean alloyed advanced SS.

The occurrence of restoration process, such as dynamic recovery (DRV) and dynamic recrystallization (DRX), in the constituent phases during thermo-mechanical processing of DSSs would significantly influence on the high temperature plastic behavior [5,6]. It has been proposed that ferrite accommodates strain preferentially because it is soft compared to austenite [7]. However, the partitioning of stress and strain in dual-phase materials is rather complex affected by several phenomena [7], such as the volume fraction of constituting phases [8] and phase morphology (size, shape, orientation and continuity of the phases) [9]. With respect to the phase fraction, some studies have clarified the effect of hot deformation parameters on hot deformation behavior of austenite and ferrite in a series of DSSs with the 50/50 phase fraction [5,10–12]. However, it must be pointed out that investigations regarding the effect of strain rate and high temperatures on the active restoration mechanisms in ferrite and austenite in DSSs with different phase fractions, for instance around 75/25 of austenite/ferrite, are very scarce. The phase ratio may affect the restoration processes at high
temperatures due to different load partitioning and fraction of $\gamma$/\(\delta\) boundaries. Moreover, in Ni-free Mn–N bearing DSSs, higher austenite fractions could be preferred since they exhibit superior room temperature mechanical properties due to strain induced martensite transformation within austenite [13–16].

DRX can occur by three different mechanisms: discontinuous dynamic recrystallization (DDRX), continuous dynamic recrystallization (CDRX) and geometric dynamic recrystallization (GDRX) [17,18]. DDRX operates by nucleation and growth and is generally considered not to take place in metals with a high stacking fault energy (SFE) because of the intense recovery, even though its occurrence is dependent on the purity of the material, e.g. Ref. [19], and on the deformation conditions, i.e. the Zener–Hollomon parameter ($Z$), i.e. the temperature compensated strain rate [20]. In CDRX, no nucleation of grains is present and new DRX grains form by the gradual increase of the misorientation of low-angle boundaries. CDRX operates at high strains 5–10, where the grains are fragmented into new ones during the deformation [21].

Austenite tends to soften through DDRX due to its relatively low SFE [19,22]. DDRX involves nucleation and growth of new recrystallized grains with the incubation time and its development with increasing strain is therefore associated with progressive softening. It is well known that DDRX grains in single-phase austenite largely nucleate via the “strain induced boundary migration” (SIBM) mechanism, utilizing the pre-existing high-angle grain boundaries [23,24]. In DSSs the availability of the pre-existing austenite grain boundaries is limited, so that the occurrence of DDRX during thermomechanical processing is restricted in austenite phase [25,26]. Consequently, austenite softens through DRV leading to the development of substructural characteristics such as microband arrays [27,28], deformation bands [29], etc. Furthermore, at high temperature regions, detailed studies have shown the austenite deformation microstructures comprising equiaxed subgrains [30,31].

In the ferrite phase the diffusion rate is about an order of magnitude higher than in the austenite phase and dislocation movement in ferrite is easier due to the higher SFE. Therefore ferrite and austenite display dissimilar behaviors during high-temperature deformation [11,20,30,32]. The dynamic restoration mechanisms in ferrite, in particular in the dual-phase microstructures, have long been the subject of intense debate among different research groups. Specifically, there are contradictory views on the effect of deformation conditions defined by the $Z$ parameter [23]. Considering its SFE values, the ferrite phase substantially undergoes CDRX during high temperature deformation. However, it can, under certain conditions, soften through DDRX [11,33]. It has been investigated that DRV (or CDRX) dominates when the Z value exceeds a certain critical threshold. Glover and Sellars [34] have reported the transition in the restoration processes from recovery at high stresses to recrystallization at low stress levels. However, there are not enough investigations about the restoration processes in DSSs with other ferrite phase fractions, concerning different accommodation of strain within the austenite and ferrite.

Finally, austenite/ferrite interfaces in DSSs affect the restoration processes during thermo-mechanical processing cycles. It has been illustrated that during hot deformation of DSSs the formation of new DDRX grains within austenite mainly occurs through the SIBM, and also partly through the subgrain growth preferentially taking place in the austenite/ferrite interface region [30]. Also, it has been observed that DDRX-like mechanism in the ferrite phase involves the formation of new grains through the growth of the highly-misoriented subgrains, preferentially formed in the interface region [11]. This is because of the unequal strain partitioning between the content phases during plastic deformation, which leads to interphase boundary sliding [25,35]. Thus, it can be concluded that the austenite/ferrite interface may have a significant contribution to softening mechanisms, but this has not been properly investigated yet.

Taking into account the industrial importance of the DSSs [1], the microstructural features and flow behavior correlations using the $Z$ parameter were investigated in more detail in the present work.

2. Experimental procedures

The hot rolled 20.55Cr-0.33N-8.21Mn-0.66Si-0.03C (in wt%) DSS was solution annealed at 1100 °C for 60 min aiming to obtain the 75/25 austenite/ferrite phase fraction. Cylindrical samples of 12 mm height and 8 mm diameter were prepared from the hot rolled bar according to ASTM E209 standard [36]. Isothermal hot compression tests were carried out at temperatures of 700, 800, 900 and 1000 °C under the strain rates of 0.001, 0.01 and 0.1 s$^{-1}$ employing a GOTECH A7000 universal testing machine coupled with a programmable resistance furnace. A thin mica foil was used to minimize the friction between the specimens and anvils during compression. For recording the applied load, a high-accuracy load cell (Model: SSMDJM – 20 kN) providing the capability of measuring the load forces down to 0.1 kN was used. To compute the true strain values, the displacement data measured from the anvils were used. Two specimens were soaked at each deformation temperature, kept for 7 min to allow the temperature to equalize throughout the specimens. One of the two specimens was quenched immediately after the annealing in order to examine the microstructure prior to the compression test, while the other specimen was deformed to the strain of 0.6 and quenched instantaneously. In order for evaluating the equilibrium phase fraction in different deformation regimes the deformed specimens were analyzed using a Ferritescope (Fischer MP30) instrument and JMatPro 6.0 software calculation.

The specimens for optical microscopy and scanning electron microscopy were sectioned parallel to the compression axis and cold mounted. These were then ground and mechanically polished. The specimens were electro-etched using a major reagent (40% solution of nitric acid at 5 V for 5 min to reveal general microstructures, austenite and ferrite). A FE-SEM ZEISS scanning electron microscope was utilized for electron backscatter diffraction (EBSD) analysis to reveal finer features of the substructures. The EBSD scans were performed at the center of the observation surface with the step sizes ranging from 0.2 to 2 μm. A TSL OIM analysis software was used for the data acquisition and post processing of the collected data. Standard method of Intercept lengths was used to calculate the average grain/subgrain sizes. The grains and subgrains were determined by adjusting the grain tolerance angle to 2° and 5°, respectively [37]. For microtexture characterization, an area of 600*400 μm with an EBSD step size of 1.42 μm, which is about 1400 grains was analyzed. Furthermore, the terms “subgrains”, “crystallites”, and “grains” relate to the microstructure units delineated fully by low angle boundaries (LABs), by both LABs and high angle boundaries (HABs), and entirely by HABs, respectively.

3. Results and discussion

3.1. Initial microstructure

Austenite and ferrite content of experimental DSS using JMatPro software is presented as a function of temperature in Fig. 1a. As can be seen, at 1100 °C the equilibrium constituent phases were estimated around 70 and 30% for austenite and ferrite, respectively, using thermodynamic calculations. Fig. 1a and b shows an EBSD phase map and grain boundary map, respectively, of the DSS in the solution-treated condition. As is seen in Fig. 1a, the microstructure consists of austenite (~75% shown by yellow color) and ferrite (~25% shown by gray color) grains with the initial sizes of 26 ± 4.4 and 25 ± 1.7 μm, respectively, which is in the range of estimated phase fraction. Furthermore, the majority of the austenite grains is occupied by annealing twins with sharp and straight Σ3 boundaries. The Kurdjumov–Sachs and Nishiyama-Wassermann orientation relationships (inside the range of $2^\circ$ deviation) for the austenite/ferrite interfaces are depicted by blue and red lines in Fig. 1b, respectively, which demonstrates a mainly incoherent nature of austenite/ferrite interfaces. The misorientation...
3.2. Flow behavior analysis

Fig. 2a displays the true stress-true strain curves obtained in hot compression tests in the temperature range of 700–1000 °C at the strain rate of 0.001, 0.01, 0.1 s⁻¹. As is observed, the DSS exhibits a considerable softening at low temperature (i.e. 700-800 °C), which may well indicate the DRX accompanied plastic flow softening under various deformation conditions; On the other hand, the softening feature at high temperature regimes (900–1000 °C) seems not drastic in comparison with low ones, and the peaks become very broad. Fig. 2b shows the effect of the deformation parameters (strain rate and temperature) on the peak stress/strain value. The peak stress and strain increase by decreasing deformation temperatures and increasing strain rates. It is well known the peak in the flow curve occurs when DRX softening mechanism dominates the work hardening behavior. As a thermally activated phenomenon, DRX is delayed as the temperature decreases, which in turn shifts the peak strain to higher ones. The diffusional character of DRX also causes a delay of the softening at higher strain rates. This may be due to the fact that less time is available for restoration to proceed at high strain rates.

The work hardening rate was predicted from the flow curves to describe the softening behavior of the DSS during hot compression. In this regard, the evolution of the strain hardening rate (θ = dσ/dε) with
true strain at different strain rates and temperatures are plotted in Fig. 3. Obviously, due to the annihilation and recombination of dislocations, the work-hardening rate decreases with the increase of strain. Moreover, the work-hardening rate is sensitive to the deformation temperature and strain rate decreasing with increase of the deformation temperature or decrease of the strain rate.

In order to consider the simultaneous influence of the temperature and strain rate, the flow behavior is interpreted adopting the $Z$ parameter defined as:

$$Z = \varepsilon \exp \left( \frac{Q}{RT} \right)$$

where $Q$ is the activation energy ($\sim 502$ kJ/mol) [38], $R$ is the universal gas constant (kJ/mol.K), $T$ is the absolute temperature (K) and $\varepsilon$ is the strain rate (s$^{-1}$). The calculated $Z$ parameters under different conditions was calculated in the range of about $1.9 \times 10^{14} - 4 \times 10^{21}$ s$^{-1}$ (Table 1). To quantify the flow softening, the flow softening index (FSI) was defined as $\left[ \sigma_{\text{peak}} - \sigma_{0.6} \right] / \sigma_{0.6}$, where $\sigma_{\text{peak}}$ is the peak stress and $\sigma_{0.6}$ is the true stress at the strain of 0.6 in different deformation conditions. Fig. 4
shows the flow softening index as a function of the $Z$ parameter. It is observed that the flow softening index decreases with decreasing the $Z$ parameter. Furthermore, the $Z$-values can be categorized into two distinct regimes, regime I (very low index, low $Z$) and regime II (high index, high $Z$) to trace the microstructural evolution of the constituent phases. According to Fig. 4, the border can be defined at $Z = 2.35 \times 10^{17} \text{ s}^{-1} (\ln Z = 40)$ for differentiating the low and high $Z$ parameter conditions. However, to figure out the corresponding mechanisms for the flow softening, microstructural characterization of ferrite and austenite is a prerequisite.

3.3. Microstructure development

Ferrite content percentages, which are essential to understand microstructural-flow behavior correlation, calculated by a Ferritoscope is represented in Table 2. As can be seen, the ferrite phase percentage remains constant. Although in stainless steels the equilibrium austenite phase fraction generally increases with decreasing temperature, in the current studying material not only did the austenite fraction not change, but it may take more time to reach the equilibrium phase fraction in different deformation conditions especially in lower deformation regimes (i.e. 700-800 °C) as well. This can be related to the fact that in engineering materials bearing high amount of alloying elements (i.e. stainless steels), the diffusion rate of alloying elements is low. Hence, the ferrite fraction may not be able to obtain its equilibrium values in the current experimental time.

To comprehend the flow softening behavior in different hot compression conditions, the microstructural evolution of constitutive phases was investigated at different temperature and strain rates. According to Fig. 5, the optical microstructures at different temperatures/strains under a strain of 0.6 show that, by increasing the strain rate and decreasing the temperature (increasing the $Z$ parameter), the

<table>
<thead>
<tr>
<th>Strain rate (s$^{-1}$)</th>
<th>Temperature (°C)</th>
<th>$Z$ parameter</th>
<th>ln($Z$)</th>
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<tbody>
<tr>
<td>0.001</td>
<td>700</td>
<td>4.01E+19</td>
<td>45.14</td>
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<tr>
<td></td>
<td>800</td>
<td>3.14E+17</td>
<td>40.29</td>
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<tr>
<td></td>
<td>900</td>
<td>5.61E+15</td>
<td>36.26</td>
</tr>
<tr>
<td></td>
<td>1000</td>
<td>1.89E+14</td>
<td>32.87</td>
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<td></td>
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<table>
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<th>Ferrite content (%)</th>
</tr>
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<tbody>
<tr>
<td>0.001</td>
<td>700</td>
<td>26.5 ± 5.1</td>
</tr>
<tr>
<td></td>
<td>800</td>
<td>27.4 ± 2.7</td>
</tr>
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<td></td>
<td>900</td>
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<td></td>
<td>1000</td>
<td>27.4 ± 2.3</td>
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<td></td>
<td>800</td>
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<td>27.1 ± 5.1</td>
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<tr>
<td></td>
<td>1000</td>
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</tr>
<tr>
<td>0.1</td>
<td>700</td>
<td>25.2 ± 2.1</td>
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<td>800</td>
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<td>1000</td>
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originally straight annealing twin boundaries in the austenite phase (light phase) transformed into distorted boundaries in the course of compression deformation (white rectangles). Furthermore, the distortion in austenite grains becomes more evident with increasing the $Z$ parameter. In addition, by increasing the deformation temperature to 1000 °C, new grains partially formed (approximately 8%) along the prior austenite grain boundaries (red rectangles). Nonetheless, it is worth mentioning that the ferrite phase (fairy darker phase) could not be revealed plainly in the optical images attributed to its sensitivity to the etching reagent. According to dual-phase steel characteristics, the flow behavior is related to both of the constitutive phases. Hence, pursuant to a high N content of the steel, along with N being an austenite stabilizer,

Fig. 5. Optical microstructure of deformed DSS specimens in the temperature range of 700–1000 °C at the strain rates of 0.001, 0.01 and 0.1 s$^{-1}$ after a strain of 0.6.
the austenite phase was harder than ferrite. It has been shown that at elevated temperatures ferrite become softer than austenite due to its higher softening rate [38]. Therefore, according to continuous morphology of the ferrite in the matrix, the plastic flow may mostly take place in the ferrite [39]. Hence, the flow behavior may be mainly determined by microstructural evolution within ferrite.

3.3.1. Region I - austenite phase

Fig. 6 present the EBSD maps of the specimens after compression at 1000 °C/0.001s⁻¹, 1000 °C/0.1s⁻¹ and 900-0.001s⁻¹ illustrating the substructural development (LABs shown by red/green lines) within austenite. A low fraction of DRX grains (about 9%, 4% and 13%) at 1000 °C/0.001s⁻¹, 1000 °C/0.1s⁻¹ and 900 °C/0.001s⁻¹ compression tests, respectively) formed near the austenite/ferrite interface (shown by arrows). Moreover, according to Fig. 7a and b, the misorientation angle distribution evolved at strain of 0.6 is characterized by a broad peak of LABs and a small peak corresponding to 60° (Σ3 boundaries). This implies the distortion of Σ3 boundaries so that some of them have lost their coherency during deformation. The relative misorientation along the linescan 1 is about 60° which can be attributed to Σ3 boundaries. Furthermore, the cumulative misorientation across the fine grains (point-to-origin) is 40° ± 20°, which is a characteristic of a random misorientation distribution [31]. This suggests that the annealing twins were largely formed during the growth of DRX nuclei, as reported elsewhere [40]. This is consistent with an assumption by Pande et al. [41]. The annealing twins are generally formed during grain boundary migration. From grain boundary serratons and bulging seen in Fig. 6a and b (shown by yellow arrows), which is the manifestation of the SIBM mechanism during deformation, it is concluded that the partial refinement of austenite grains occurred by the DRRX mechanism. On the contrary, cumulative misorientation across line 2 shows a complex trend and values typical of HABs, even though the developed sub-boundaries are characterized by low-angle misorientations (point-to-point misorientations in Fig. 7c). A similar structural feature has been observed in early studies of the CDRX mechanism [31].

3.3.2. Region I - ferrite phase

Fig. 8a–f illustrate the ferrite microstructure of three specimens processed at 1000 °C/0.001s⁻¹, 1000 °C/0.1s⁻¹, and 900 °C/0.001s⁻¹ after a strain of 0.6. As can be seen (Fig. 8a–d), large equiaxed grains developed besides subgrains with an average grain sizes of 18 and 15 μm in the 1000 °C/0.001s⁻¹ and 1000 °C/0.1s⁻¹ specimens, respectively. However, by decreasing deformation temperature, the grain size reached to 10 μm in 900 °C/0.001s⁻¹ specimen. These results indicate that many LABs were formed within the grains in the early stage of deformation, and some LABs gradually transformed into HABs with prolonged deformation. This demonstrates that the grain refinement of ferrite under these deformation conditions occurred by the CDRX mechanism by transforming subgrains into new grains although there are differences in transformation rate of subgrains to grain at 1000 °C/0.001–0.1s⁻¹ and 900 °C/0.001s⁻¹ specimen.

Fig. 9 displays the ferrite/austenite interface region formed at 1000 °C/0.001s⁻¹ in compression to a strain of 0.6. The KAM map with the misorientation between 0 and 5° represents the presence of high dislocation density regions forming LABs adjacent to the austenite/ferrite interface (Fig. 9a). The misorientation profile near the ferrite/austenite interface (linescan 1) shows the subboundary formation with a larger misorientation (see Fig. 9b) and displays significantly larger cumulative misorientation gradients compared to the gradients in the ferrite matrix (linescan 2). This means higher strain accumulation within the interphase boundary regions. As can be seen, the point to point misorientation close to the interface (linescan 1) is about 1.5° which is larger than that of LABs formed within the ferrite phase (about 0.6° along linescan 2). In addition, the point to origin misorientation gradient near the interface is higher than farther region. This can be attributed to interface boundary sliding between the austenite and ferrite phases, which creates a misfit strain between ferrite and austenite [30]. The sliding is expected to be promoted by the present largely non-coherent character of the interphase (Fig. 1a).

3.3.3. Region II - austenite phase

EBSD maps shown in Fig. 10 illustrate the developed substructures in the austenite phase in the course of compression at 800 °C/0.001s⁻¹, 800 °C/0.1s⁻¹ to a strain of 0.6 in the region II. The corresponding diagrams of the misorientation angle distribution are shown in Fig. 11a and b. Similar to region I, the misorientation angle distribution in these deformation condition is characterized by an evident peak of LABs at about 4° and a small peak corresponding to 60° (Σ3 boundaries). This can be related to the distortion of Σ3 boundaries which some of them lost their coherency during compression. As seen in the EBSD maps in Fig. 10a and b, new parallel sub-boundaries (2–5° and 5–15° boundaries represented by red and green lines, respectively) formed inside grains, which were characterized as microbands. These microbands appeared in various original grains and were accompanied by the development of a characteristic orientation gradient within a deformed grain, seen in an IF map in Fig. 10c and d. The cumulative misorientation profile along these parallel sub-boundaries (linescan 1) is typically more complex and shows a frequent accumulation of misorientations along significant distances (Fig. 11c). The point to origin misorientation profile across the microbands (linescan 2) generally demonstrates systematically alternating misorientations indicating that the misorientation variations across consecutive extended walls tended to cancel each other (Fig. 11c). Additionally, as is seen from Fig. 10c and d, in some regions strain induced boundaries, shown by black lines, formed in the microband sections. It is worth mentioning that some of LABs exhibited medium misorientations of 5–15° (Fig. 10a), which can be potentially considered to evolve into HABs. Moreover, some subboundaries, which are mostly formed near austenite/ferrite interfaces, show large accumulation of misorientation locally up to about 20° (misorientation linescan 3 in Fig. 11c). Furthermore, as is evident from Fig. 10c, austenite grains were broken in mutually rotated complex-shaped fragments that are separated by a transition region.

As investigated at high Z values, substructure was developed within austenite phase which is considered as DRV restoration process. This cannot be considered as flow softening factor in regime I (Fig. 4). Thereby, microstructural evolution within ferrite is necessary to investigate in order to characterize the flow softening behavior, precisely.

3.3.4. Region II - ferrite phase

A grain boundary map, IPF map, and kernel average misorientation (KAM) image illustrating the ferrite microstructure of a specimen, compressed at 700 °C under the strain rate of 0.001s⁻¹ to a strain of 0.6, are shown in Fig. 12. The HABs are labeled by blue lines whereas the LABs (<15°) are red and green lines (Fig. 12a). As is seen, the grain size was decreased in the ferrite phase (to about 5 μm), where new segments containing subgrains with LABs were surrounded by HABs (strain-induced boundaries represented by blue color). These segments called DRX grains are illustrated by white arrows in the boundary and IPF map (Fig. 12a and b). As can be seen, grain boundaries consisted of induced LABs besides LABs, indicating that they were formed during straining. Based on the LAB/HAB fraction above 50%, it can be deduced that grain refinement of ferrite in this condition has occurred by the CDRX mechanism [20]. Regarding to Fig. 12b, the low Kernel average misorientation values (blue color) indicating a low density of dislocations support the idea that the microstructural evolution accompanied by extended recovery or recrystallization [42].

By increasing the test temperature to 800 °C under a strain rate of 0.001s⁻¹, DRX grains within the ferrite phase became more discernible (Fig. 13a). This means that the CDRX process further developed compared to that of compressed specimen at 700 °C/0.001s⁻¹ since ferrite phase under this deformation condition recovered (annihilation and rearrangement of dislocations) at a higher rate. Also, the KAM map
Fig. 6. EBSD analysis of austenite in TMP DSS at (a, b) 1000 °C/0.001 s⁻¹, (c, d) 1000 °C/0.1 s⁻¹ and (e, f) 900 °C/0.001 s⁻¹ at a strain of 0.6. (a, c, e) Corresponding boundary maps and (b, d, f) IPF maps of austenite phase.
illustrates low dislocation densities within the ferrite phase (blue region, Fig. 13), which supports the fact that the microstructural evolution occurred by recrystallization [42]. The misorientation profile along the line A-B shows that point to point misorientations are between 10 and 60°, which depicts that HABs evolved to LABs during straining. Furthermore, the cumulative misorientation profile as a function of distance alternates and displays a complex trend of HAB misorientations in the ferrite phase. The cumulative (point-to-origin) misorientation attains typical values of HABs although the developed deformation sub-boundaries were characterized by low-angle misorientations (point-to-point misorientations in Fig. 13g). Similar substructural characteristics have been observed in early stage of CDRX [31]. It has been suggested that the transition of LABs into HABs, and consequently the CDRX rate, can be controlled by a recovery rate, which is accelerated by the increasing deformation temperature [43]. Hence, subgrains can simply form new grains by rotation or transforming a LAB to a HAB. Furthermore, in the case of the higher strain rate (800 °C/0.1s) more dislocations are absorbed into high angle boundaries since there are more blue regions comparing KAM maps in Fig. 13c and f. This can be related to the fact that at high strain rate the dislocation density increases.

In order to investigate the substructure evolution (i.e. subgrains segments formation) during deformation in this deformation regime interrupt test was performed. Fig. 14 illustrates the microstructural feature inside the deformed ferrite matrix compressed at 800 °C under a strain rate of 0.001s⁻¹ to a strain of 0.2. As can be seen in Fig. 14a, substructures with irregular shapes enclosed by low to medium angle boundaries were formed within ferrite. Besides, various strain induced subboundaries formed near austenite/ferrite interface boundaries. This is due to the interface boundary sliding mechanism where high strains are induced near interphase boundaries to accommodate deformation. Furthermore, some isolated segment containing various subgrains possessing nearly crystallographic orientations were formed (shown by black arrows in Fig. 14b). Different subgrains and segments can be differentiated distinctly by their respective orientations. Examination of local orientation relationships of the subgrains also displays that they demonstrate a near orientations within segments (1, 2, 5 and 6 points) and wide arc spanning (3, 4 in comparison with 7) in corresponding IPF (Fig. 14c). This suggests that subgrains within segments formed in the first stage of compression further misoriented in the following straining, such that they are assisted to organize themselves to form grains (Fig. 13a). Accordingly, it is further proof that CDRX mechanism under this present deformation condition occurred.

EBSD images of the ferrite phase of a specimen compressed at a higher temperature and strain rate (900 °C-0.1s⁻¹) to the strain of 0.6 are shown in Fig. 15. As seen, the microstructure has a distinct feature in comparison with other conditions where the CDRX was the dominant restoration mechanism. The subgrains surrounded partly by LABs and HABs were rarely formed within ferrite. However, new grains free of subgrains formed near the austenite/ferrite interface boundaries (illustrated by a white arrow in Fig. 15a). It seems that these grains nucleated near the interphase boundaries and consequently grew in order for annihilating strain energy. The KAM map in Fig. 15c illustrates the strain-free grains (blue colors) growing into high strain energy regions where aggregates of dislocated subboundaries are shown by green lines (pointed by a black arrow). The microstructure appears as a combination of fresh grains without substructure and deformed grains consisting of subgrains. Thus, we can conclude that in this condition instead of the CDRX, the DDRX mechanism predominantly occurred in the ferrite phase. Even though ferritic alloys have high SFE, it seems that the activation of the DDRX mechanism under certain deformation conditions is possible. Generally, in the DDRX mechanism, the dynamic recovery process is less effective since strain rates are relatively high; thus, the duration of the tests is limited. This results in higher strain hardening rate which promotes local accumulation of dislocations in the ferrite phase. As a result, the nucleation occurs by forming a subgrain
Fig. 8. EBSD analysis of ferrite in TMP DSS at (a, b) 1000 °C/0.001s⁻¹, (c, d) 1000 °C/0.1s⁻¹ and (e, f) 900 °C/0.001 s⁻¹ at a strain of 0.6. (a, c, e) Corresponding boundary maps and (b, d, f) IPF maps of ferrite phase.
surrounded by HAB at high strain regions (interface regions and ferrite/ferrite boundaries) and growth takes place by moving the grain boundary to high stored energy regions. This can clarify the occurrence of DDRX mechanism under the medium Z parameters conditions in the present study.

The schematic illustration of the DRX mechanisms in the present DSS is shown in Fig. 16. In low and high Z parameters, the substructure development within ferrite characterized by a gradual increase of crystallite misorientation during straining accompanied by the progressive conversion of the original subgrains bound by LABs to (sub) grains delineated partly by LABs and partly by HABs. The process leading to the formation of such pronounced hierarchy of low and high angle boundaries might be classified as CDRX reported to operate during hot deformation of ferrite phase in both single-phase [31] and duplex steels [44]. Gourdet et al. [45] have proposed a model of the CDRX process that occurs in two ways, i.e. by the condensation of dislocations into new LABs, and the absorption of dislocations in the pre-existing boundaries, which leads to the transformation of LABs to high angle ones. Thus, CDRX typically results in the formation of new roughly equiaxed “crystallites” bounded partly by LABs and partly by HABs. Oudin et al. [46] have attributed the formation of HAB segments observed during hot torsion in a Ti-IF steel to the accelerated rotation of isolated subgrains occurring due to variations in the crystallographic rotation field and/or to the establishment of different configurations (selection or amplitude) of slip systems in neighboring regions. As is seen in Figs. 12 and 13, some segments consisting subgrains, which were separated by HABs, formed DRX grains. Haghdadi et al. [11] have demonstrated that such groups of subgrains rotated over large angles from the surrounding matrix bear some resemblance to deformation bands.

Because of a high SFE of the ferrite phase, it is expected that the CDRX mechanism might take place during hot deformation of ferritic steels. It has been reported that ferrite in both single-phase and dual-phase steels might soften through CDRX under wide thermo-mechanical conditions [47,48]. Castan et al. [20] applied hot torsion to deform a ferritic low-density steel. They concluded that at a high temperature and low strain rate, material undergoes CDRX. However, under certain conditions, CDRX may be hindered and another softening mechanism such as DDRX comes into operation. In dual-phase alloys such as DSSs with the equal phase fraction of ferrite and austenite it has been concluded that a significant increase in the strain rate (i.e. in the Z) resulted in the change of the ferrite softening mechanism from CDRX to a distinct DDRX mechanism [11]. Anyway, in this study it is concluded that in the current experimental DSS with two phase structure, the DDRX softening mechanism occurred at lower temperatures and strain rates (900°C-0.1s⁻¹), though this mechanism has occurred in higher strain rates and temperatures (i.e. 1100°C/5-10s⁻¹ for a ferritic steel in the study of Castan et al. [20] and 1000°C/10s⁻¹ for a dual-phase alloy in Haghdadi et al. [11] work with the equal fractions of ferrite and austenite. This may be related to higher strain partitioning within the softer phase (ferrite) in present investigated material. In DSSs, the soft ferrite phase tends to accommodate the strain during hot deformation cycles [49]. Hence, it can be concluded that by decreasing the ferrite volume fraction, local strain rate within the ferrite phase is higher than that in the fully ferritic matrix. Consequently, in the present experimental material with the low ferrite fraction, a higher strain and accordingly higher strain rate was applied to the ferrite phase. Furthermore, the lower fraction of ferrite is in DSSs microstructure, the more γ/δ interface boundaries exist for nucleation of DDRX grains. It is worth mentioning that in this experimental DSS there is a high fraction

Fig. 9. (a) Kernel average misorientation (KAM) map of deformed ferrite phase corresponding to 1000°C/0.001s⁻¹/0.6 specimen. (b) Misorientation profile (point to point and point to origin) along the linescan 1 and 2 showing high strain area near interphase region.
of $\gamma/\delta$ interface boundaries according to low ferrite fraction. So, the DDRX mechanism was induced at low strain rates under conditions where the mobility of boundaries is fairly high for the growth of new nuclei (Fig. 15).

3.4. Evolution of austenite/ferrite substructure under various Z conditions

3.4.1. austenite phase

To understand the evolution of twin boundaries and substructure within austenite during compression, the fraction of LAB, HAB, $\Sigma 3$ boundaries were calculated from EBSD data at the strain of 0.6 (Fig. 17). It can be seen that the $\Sigma 3$ boundary fraction were decreased with increasing $Z$ parameter without linear relationship where the maximum value (~0.2) is at 1000 °C/0.1s$^{-1}$. Thus, it can be deduced that the origin of these $\Sigma 3$ boundaries are the pre-existing annealing twin boundaries in the solution annealed material. The variation of $\Sigma 3$ boundaries in different conditions shows a sluggish evolution of these boundaries (maximum fraction of about 0.2) in different $Z$ parameters.
Fig. 11. (a, b) Corresponding misorientation angle distribution in Fig. 10a and b, respectively. (c) Misorientation profile along the linescan 1–3 superimposed in the structure in Fig. 10a.
Fig. 12. EBSD analysis of the microstructure in the ferrite phase deformed at 700°C/0.001 s⁻¹ to a strain of 0.6: (a) grain boundary map, (b) IPF map, (c) KAM map.
Fig. 13. EBSD analysis of the ferrite phase, compressed at (a–c) 800 °C/0.001s⁻¹ and (d–f) 800 °C/0.1s⁻¹ to a strain of 0.6. (a, d) Boundary map, (b, e) IPF map and (c, f) KAM map between 0 and 5°. (g) Misorientation profile along the black arrow superimposed in (a).
This is due to the fact that the contribution of DRX grains for \( \Sigma 3 \) boundaries formation is extremely low since low fraction of DRX grains formed even in favorable conditions (maximum fraction of 13% at 900 °C). In other words, owing to a lack of the pre-existing grain boundaries within austenite, the occurrence of DRX and, subsequently, \( \Sigma 3 \) boundaries are limited in DSSs [25,26].

Moreover, with increasing \( Z \) parameter, deformation severity increased and more pre-existing \( \Sigma 3 \) boundaries may lose their coherent character in the course of compression. To clarify this explanation, \( \Sigma 3 \) boundaries deviation also calculated from EBSD data according to Brandon criterion [50]. As can be seen the deviation degree from ideal \( \Sigma 3 \) boundaries increases at higher strain rates and lower temperatures (from 3 to 5.7°) showing these special boundaries are rarely free of deformation in different \( Z \) parameters. The high contribution of LABs
during hot compression of austenite (~0.5–0.9) shows that austenite undergoes limited DRX and DRV entirely takes place during hot compression. This also illustrates that most of the Σ3 boundaries are hardly formed during hot compression. Otherwise, the average deviation of Σ3 boundaries would be decreased by imposed deformation.

Fig. 15. EBSD analysis result of the ferrite phase in specimens deformed at (a–c) 900 °C/0.1s⁻¹ to a strain of 0.6. (a) Grain boundary map, (b) IPF map and (c) KAM map between 0 and 5°.
Fig. 16. Schematic illustration of different DRX mechanisms within the ferrite phase at different $Z$ values.

Fig. 17. Deviation from ideal $\Sigma 3$ boundaries and variation of fraction of $\Sigma 3$ boundaries, LAB and HAB under different $Z$ parameters to the strain of 0.6.
3.4.2. 4. ferrite phase

The occurrence of CDRX and DDRX mechanisms can be illustrated in a quantitative manner by misorientation angle distribution of boundaries within the ferrite phase (Fig. 18). At high Z parameters (700–800 °C/0.001 s⁻¹), where the dominant softening mechanism was CDRX according to microstructural characterization, a large fraction of LABs (about 70%) are formed. Furthermore, at low Z parameters (1000 °C/0.001 s⁻¹), the average misorientation angle is 10.3° indicating that LABs may need more strain to transform into HABs. This shows DRV mechanism is more pronounced in this deformation condition although some CDRX grains formed within ferrite matrix (Fig. 8). Conversely, at medium Z parameter (900 °C/0.1 s⁻¹), the misorientation histogram shows a high contribution of HABs within ferrite (about 75%). Indeed, the distribution of misorientation angle is quite different in the conditions where the DDRX mechanism operated and is similar to the Mackenzie random distribution [51]. This must be related to the active softening mechanism in this condition, where high-angled subgrains nucleated at high strain regions and grew in order to eliminate strain energy fields.

The microtexture evolution within ferrite of the experimental DSS during the hot compression where the CDRX (800 °C/0.1 s⁻¹) and DDRX (900 °C/0.1 s⁻¹) mechanisms were dominant is represented with IPF maps normal to the compression axis in Fig. 19. The microtexture of the ferrite prior to the hot deformation reveals the random character with weak crystallographic texture (about 1.475) at the direction of <001>. However, after hot compression at 800 °C under the strain rate of 0.1 s⁻¹ and a strain of 0.6, the crystallographic texture was identified by the continuous crystallite rotation in the directions of <111> and <100>, which are noted to be the stable end orientations under uniaxial compression of bcc metallic materials [23]. This is consistent with the literature [52,53], texturizing toward the stable directions occurs when the CDRX restoration process was active during deformation. On the other hand, at 900 °C/0.1 s⁻¹, where DDRX mechanism occurs, the texture intensity fairly decreased compared to the primary microstructure at <001> direction. Indeed, the texture of the deformation matrix containing subgrains was analogous with that obtained at the lower
temperature. Meanwhile, by formation of DRX grains through discontinuous mechanism the number of texture components is decreased and thus the dominated <001> fiber is decreased toward random character obtaining the texture intensity of 1.26. These results collaborate the findings of a great deal of the previous works in this field [11, 23]. This is a thriving result limiting texturized orientation by inducing DDRX mechanism in order for obtaining random orientations.

In order for providing insights into the relation between DRX evolution within ferrite and flow softening behavior, quantification of ferrite (sub) structure was needed. There are different methods to determine the DRX fractions in hot compressed specimens such as measurement of grain size, KAM and grain orientation spread (GOS) [54]. The GOS of a grain is defined as the average of difference in orientation between the average grain orientation and the orientation of each scan point within that grain, whereas the KAM is described within a kernel instead of a grain. Generally, the GOS approach depends on the average value of this parameter, which should be lower for DRX grains than deformed grains. For differentiating the DRX grains from the deformed grains, a cut off criterion has been defined, according to Mandal et al. [55] the ‘‘1’’ cutoff value has been determined to distinguish between DRX and deformed grains in austenitic steels. The GOS approach was a suitable method to partition DRX grains formed through nucleation and growth mechanisms (DDRX) [56]. Nevertheless, it should be noted that in CDRX, new grains become deformed due to continuous deformation, so that they are free of applied strain only very shortly.

Fig. 20 shows the distributions of GOS and KAM within the ferrite phase in different deformation conditions after the strain of 0.6. Clear peaks in the misorientation values for all the specimens can be noticed. Considering the GOS and KAM values, which are shown in Fig. 20 a and b, respectively, for specimens deformed at high Z parameters (i.e. 700–800 °C) both GOS and KAM plots show broad peaks between 3 and 15°. These characteristics might be a result from the presence of a high fraction of CDRX segments containing subgrains. On the other hand, at 900 °C/0.1s−1, GOS and KAM values show distinct peaks in the low range of 1–2°, which can be due to the presence of new fresh grains formed through DDRX mechanism.

As seen in Fig. 20a, which is related to specimens deformed in the high Z parameter region (i.e. 700 °C/0.001s−1), there are multimodal distributions of GOS. As mentioned before, the ferrite phase softens through the CDRX mechanism, which is a continuous restoration phenomenon where subgrains transform to new grains. Thus, the multimodal distribution of GOS can be related to the co-existence of crystallites and (sub)grain segments. Nevertheless, at 900 °C/0.1s−1 the ferrite softening mechanism turns to DDRX, where high-angle subgrains transform into nuclei followed by growing through high strain regions.
Consequently, fresh grains substituted for the deformed matrix and intense peak with lower GOS (<1.5°) appeared. In theory, GOS is the average difference in the orientation between the average grain orientation and all measurements within a single grain. In this regard, GOS represents the orientation gradient of grain interior and its increase is generally considered as the result of increasing heterogeneity during deformation. Such an orientation gradient is usually accommodated by dislocation multiplication and dislocation cell refinement [57]. So, the formation of free-strain grains as a result of growth of high-angle subgrains leads to diminishing elastic energy within the deformed matrix.

Additionally, Table 3 illustrates the mean values of these parameters in various Z parameters. As is shown, the mean values of GOS were about 3° and 1.5° for specimen undergone CDRX (Figs. 8 and 13) and DDRX (Fig. 15) mechanisms, respectively. So, in this study, the mentioned cutoff values were proposed to investigate DRX progress, calculating non-deformed grains fraction in different Z values. It is worth mentioning that CDRX grains have more orientation spread in comparison with DDRX ones. This can be related to the fact that in CDRX grains, difference in orientation between the grain and all measurements in a grain increases because of the subgrain-base formation of new grains. However, DDRX grains are almost free of subgrains since new boundaries tends to move in order for eliminating deformed regions.

Fig. 21 represents a diagram of subgrain, grain sizes calculated by average intercept length method besides non-deformed DRX grains (N-D DRX) fraction as a function of the Z parameter. In DDRX dominant regions, non-deformed DRX grains referred to the crystallites which were considered to be almost minimum in orientation spread. As is shown, with decreasing Z values subgrain/grain sizes increases in a logarithmic linear relation. This can be related to the reason that at lower Z parameters (high temperatures) lower dislocation densities and stored energy accumulated during deformation. On the other hand, at Z > 3*10¹⁹ the subgrain/grains sizes which were formed through CDRX mechanism reached to 4.5/4.9 μm, respectively. This is because higher stresses were imposed, so that large fraction of dislocations in subgrain configuration formed to accommodate strain throughout structure. Additionally, the volume fraction of N-D DRX grains calculated by GOS measurement increases by increasing Z values with a nearly linear trend in the logarithmic Z scale. At high Z parameters (4*10¹⁹) the fraction of N-D DRX grains reached to 65% which can be due to the presence of fine crystallites. The GOS measurement does not consider CDRX segments consisting of subgrains as non-deformed DRX grains since they would need larger strains to transform into non-deformed DRX grains (crystallites) bearing sizes similar to subgrains in that deformation condition. With decreasing Z parameter to 5.6*10¹⁷ (900 °C/0.1 s⁻¹), the fraction of N-D DRX grains formed through DDRX mechanism reached to about 70%. On the other hand, at Z = 1.9*10¹⁴ in region I, the fraction of N-D DRX grains decreased to 20%. This can be due to the fact that at high temperatures (1000 °C) DRV is more pronounced since limited low angle boundaries had tendency to transform to CDRX grains (Figs. 8 and 18).

As mentioned in previous sections, austenite softens through DRV mechanism in almost all Z parameters, except that in region I a very low fraction of newly formed DRX grains (up to a maximum of about 12%) which did not lead to an apparent decrease in flow curves (Figs. 2 and 6).

### Table 3

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>700</th>
<th>800</th>
<th>900</th>
<th>1000</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strain rate (s⁻¹)</td>
<td>0.001</td>
<td>0.001</td>
<td>0.1</td>
<td>0.001</td>
</tr>
<tr>
<td>Mean GOS (degree)</td>
<td>3.2</td>
<td>2.4</td>
<td>1.9</td>
<td>2</td>
</tr>
<tr>
<td>Mean KAM (degree)</td>
<td>1.01</td>
<td>0.88</td>
<td>0.55</td>
<td>1.06</td>
</tr>
</tbody>
</table>

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Fig. 20. (a) GOS and (b) KAM charts corresponding to the ferrite phase for specimens deformed at 700, 800, 900 and 1000 °C with the strain rates of 0.001 and 0.1 s⁻¹ to a strain of 0.6.

Fig. 21. Diagram of grain/subgrain sizes and the volume fraction of non-deformed DRX grains calculated through GOS measurements within ferrite as a function of Z parameter after the strain of 0.6.
Comparing the N-D DRX fraction in Fig. 21 with FSI values in Fig. 4, it can be concluded that the fractions of the new strain-free grains within ferrite are almost correlated with flow softening behavior. It is expected that during DRX mechanism, the flow stress decreases during straining because of the restoration processes dominate work hardening rate. For instance, in this study at extremely high Z parameters (lnZ ~ 45) in region I, the N-D DRX fraction reached a level of about 65% which justifies the higher FSI values shown in Fig. 4. On the other hand, at low Z parameters in region II, the FSI was near to 0 showing the DRV may probably have contribution to induce steady state character in flow curves. As was seen in ferrite phase (Figs. 8 and 21), large subgrains/grains alongside with a low fraction of DRX grains were the main reason for limited FSI values in flow curves.

4. Conclusions

The substructure development and restoration processes during hot compression of a DSS with the 75/25 austenite/ferrite ratio was investigated at various temperatures and strain rates. The following main conclusions can be drawn:

1. DRV was the dominant softening mechanism in the austenite phase under various conditions by forming different substructure characteristics (microbands and transition regions). Microband arrays had a specific character resembling to band-like misorientation gradients. Only a low fraction of DRX grains (a maximum value of 13%) formed through CDRX/DDRX mechanisms even at high temperatures because of intense stress and strain concentration within the ferrite phase during deformation of DSS.

2. In the ferrite phase, the CDRX softening operated at high Z parameters (Z~10^19-10^20). It was observed that within ferrite some subgrain segments formed in the beginning of the straining, which content subgrains transformed to non-deformed crystallites resulting CDRX mechanism.

3. At the medium Z conditions (Z ~ 5.6*10^17 s^-1) the DDRX mechanism was the dominant restoration process. This mechanism incorporates in the formation of new grains through the growth of highly-misoriented subgrains, preferentially at the ferrite/austenite interface phases. This mechanism was identified in the ferrite phase in the lower strain rate regime (0.1 s^-1) in comparison to the strain rates reported in the literature, because of highly available γ/δ interface boundaries as well as high strain accommodation of ferrite.

4. In this study a GOS cutoff values of “1.5” and “3” were proposed where CDRX and DDRX mechanisms were dominant in ferrite phase, respectively.

5. With decreasing Z parameters, the contribution of DRX to ferrite grain refinement increased, so that the volume fraction of DRX increased with a uniform trend. This is due to that low temperatures and high strain rates boosted stored energy of deformation which drives the refinement process inside the original grains. Moreover, at low Z parameters the sub-boundaries fraction increased by the decreasing Z parameter showing DRV dominates over CDRX mechanism as well as the size of the subgrains increases with increasing deformation temperature.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

H. Khatami-Hamedani: Conceptualization, Methodology, Software, Writing - original draft, Visualization, Investigation, Data curation, Formal analysis, Writing - original draft. A. Zarei-Hanzaki: Supervision. H.R. Abedi: Validation, Investigation, Writing - review & editing. A.S. Anouche: Investigation, Writing - review & editing. L.P. Karjalainen: Investigation, Writing - review & editing.


